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QUANTITATIVE METALLOGRAPHIC ASSESSMENT OF HIGH TEMPERATURE COMPONENTS

Report No 87-267-K

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ABSTRACT

Surface replication is an important tool used in life assessment of thermal units. The immediate application is damage/defect detection but it can also be used to characterize and quantify more subtle changes in the microstructure of low-alloy steels, changes which weaken the material and lead ultimately to failure. Quantitative metallographic techniques based on replication have been used to estimate metal temperatures in selected locations of several Lakeview units. The report analyses the methods and results in some detail and identifies areas where work is needed. Also considered is the potential of estimating creep strain from microstructural studies. To obtain both temperature and strain in this non-destructive way would be very valuable in terms of life assessment. Because of the potential benefits, it is recommended that a specific research program on quantitative metallographic methods should be formulated and appropriately resourced.

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EXECUTIVE SUMMARY

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Surface replication is a major tool in the life assessment work presently being conducted on critical components of thermal units. The immediate purpose is to detect evidence of creep (cavities, micro-cracks) or to characterize other forms of damage which may or may not have been disclosed by visual or non-destructive examination. Replication can, however, also facilitate the characterization and quantification of more subtle changes which occur in the microstructure of low alloy steels in elevated temperature service, changes which weaken the material and which would ultimately lead to failure.

One change which occurs is the coarsening of carbide particles in the microstructure and the concomitant increase in interparticle spacing. This is a thermally-activated process and so, given appropriate data pertaining to coarsening kinetics, affords a means of estimating the effective metal temperature of the sampled component location. Since operating temperature strongly influences creep life, this provides a vital input to life assessment. Temperature estimations using this approach have been made for Lakeview Unit 1 components and latterly for other units.

This report describes the techniques used to obtain the quantitative microstructural data (mean particle size and spacing) then considers various aspects in some detail, firstly to assess the degree of confidence to be placed on the results and, secondly, to identify areas where more work is needed.

Another related but less developed application is to relate quantifiable microstructural parameters to the creep strain in the sampled location. Some of the present data have been input to published work in this area (some empirical, some theoretical) and have yielded interesting results.

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The potential thus exists for non-destructively obtaining from a sampled location estimates of both metal temperature and creep strain, a powerful combination in terms of life assessment and remaining life prediction.

Because of the potential value of the approach, it is recommended that a specific research program on quantitative metallographic methods be formulated and the necessary funding and resources made available.

TABLE OF CONTENTS

	Page
LIST OF TABLES	iv
LIST OF FIGURES	iv
1.0 INTRODUCTION	1
2.0 EXPERIMENTAL PROCEDURES	2
2.1 Surface Replication	2
2.2 Carbon Extraction of Particles	2
2.3 Particle Size Analysis	2
3.0 RESULTS	3
4.0 ANALYSIS OF DATA - METAL TEMPERATURE ESTIMATION	3
4.1 Estimation Based on Mean Particle Diameter	3
4.2 Estimation Based on Interparticle Spacing	4
5.0 DISCUSSION	5
5.1 Use of Inappropriate Coarsening Data	6
5.2 Significance of Original Particle Size	6
5.3 Interparticle Spacing Values	7
5.3.1 Mean Free Path	9
5.4 Possible Error in Particle Spacing Value	10
5.5 Temperature Estimation Using Interparticle Spacing	10
5.6 Relating Interparticle Spacing to Creep Strain	11
6.0 SUMMARY AND CONCLUSIONS	13
7.0 RECOMMENDATIONS FOR FUTURE WORK	14
8.0 ACKNOWLEDGEMENTS	15
REFERENCES	16
TABLES	17 - 22
FIGURES	23 - 29
APPENDIX - ONTARIO RESEARCH FOUNDATION REPORT	30
DISTRIBUTION	last page

LIST OF TABLES

Table		Page
1	Quantitative Metallographic Data - Lakeview Unit 1	17
2	Metal Temperature Estimations from Quantitative Metallography	19
3	Temperature Estimates for 1-1/4 Cr-1/2 Mo Components	21
4	Temperature Estimates for 2-1/4 Cr-1 Mo Using Williams & Parker Data	22

LIST OF FIGURES

Figures		Page
1	Replica Washing Apparatus	23
2	Typical T.E.M. Micrographs	24
3	Williams Coarsening Rate Data	25
4	Comparison of Williams and Parker Coarsening Results	26
5	Plot Showing Sensitivity of d_t to d_o	27
6	Hale Results Relating Particle Spacing to Creep (a) Creep Strain, (b) Creep Strain Rate	28
7	Remaining Needle-like Particles in 2-1/4 Cr-1Mo Samples	29

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To Dr. D. Mills
Director of Research

QUANTITATIVE METALLOGRAPHIC ASSESSMENT OF HIGH TEMPERATURE COMPONENTS

1.0 INTRODUCTION

One of the contributions of Metallurgical Research to the current Life Assessment Program on thermal units is the metallographic examination of major components in the boiler, turbine and high energy piping systems using techniques of in-situ surface replication. The immediate purpose of this work is to detect evidence of creep damage (cavities, micro-cracks) or to characterize other forms of damage (casting defects, fatigue cracks, etc) which may or may not have been disclosed by visual or ND examination. The results of this work on Lakeview Unit 1 have been reported/1/, work on Unit 4 is presently near completion and work on Unit 7 is just getting underway.

In addition to the detection of structural discontinuities, however, surface replication can also facilitate the characterization of the more subtle changes which occur in the microstructures of low alloy steels exposed for long periods at elevated temperatures (> ~450°C). These changes essentially involve the coarsening and compositional changes of the carbide particles which are largely responsible for the creep resistance of these materials. Coarsening can be quantified by determining average particle size and/or interparticle spacing which can be done fairly readily using modern Image Analysis systems. The data obtained can then, in principle, be analyzed in terms of coarsening kinetics to yield an estimate of the effective metal temperature for the component location; this then affords a powerful tool to be used in the life assessment of the particular component. Also, quantitative data of this type provides a valuable "fingerprint" of the state of the unit for potential comparison when a second, more stringent life assessment is carried out at some future date.

Results of quantitative work on Lakeview Unit 1 were supplied in memorandum form in June 1987 /2/. This present report describes the work in more detail, including the practical aspects, analytical approaches and also some additional work performed subsequent to the initial reporting.

2.0 EXPERIMENTAL PROCEDURES

2.1 Surface Replication

Surface preparation and acetate replication were carried out using the procedures fully described elsewhere/3/. Grinding and polishing down to 1 μm were followed by an initial light etch in 5% nital. The surface was replicated and then a second etch was applied and a second replica produced.

Most of the replicated locations were at the site of welds, since creep damage is particularly expected in coarse-grain heat affected zones. For the quantitative metallographic assessment, however, it was necessary to examine parent material with no structural modification due to welding. Accordingly, the areas selected were always taken at the furthest point on the replica away from the weldment.

2.2 Carbon Extraction of Particles

The level of magnification needed for detailed examination of the fine carbide particles in low alloy steel microstructures dictates the use of the transmission electron microscope, either on thin foils with the particles contained or on carbon extraction replicas. Clearly, the non-destructive approach of in-situ replication precludes the use of thin foils. For present purposes, therefore, carbon extraction was used on the replicas and, to ensure the best degree of particle extraction from the component microstructure, acetate replicas from the second etch stage were used.

The replicas were carbon-coated using an Edwards vacuum evaporator and were then cut into 3 mm squares which were placed on copper grids. Removal of the cellulose acetate was then accomplished by means of the vapour washing method, using the apparatus shown in Figure 1. The copper grids were placed on a 200 mesh screen wired securely to a water-cooled glass stage shown at A.

The acetone solvent was heated and vapours condensed directly on to the replica on the cold stage. After approximately two hours the acetate backing was completely dissolved leaving the carbon film on the copper grid. The grid and carbon film were then ready for examination which was performed in a JEOL 100C transmission electron microscope.

The vapour washing method described above can produce a relatively large area from the replica without rupture or distortion. Some alternative methods, although quicker, can seriously strain the film, to the extent that much of it may be effectively destroyed.

2.3 Particle Size Analysis

Representative electron micrographs were taken of the carbon extraction replicas at a standardized magnification of 10 000X. This afforded adequate resolution of fine particles together with a sufficient number for statistically-meaningful analysis. Figure 2 shows some typical micrographs.

In selecting areas for the micrographs, grain boundaries were avoided since it is only particles within the grains which are normally included in coarsening determinations/4/.

The micrographs were subjected to image analysis using a LeMont OASYS system at the Ontario Research Foundation (ORF). Full details of this work are given in the appended report which is reproduced with the kind permission of ORF.

3.0 RESULTS

Results of particle size analyses on replicas from boiler, turbine and high energy piping locations are presented in Table I. Also included are the replica ID numbers which can be used to obtain specific details of sample locations, if required. As described in the ORF report, the particle mean diameter, deviation and median values were determined directly by the OASYS system; the mean interparticle spacing values id were determined using the simple relationship

$$id = \left(\frac{A}{N} \right)^{1/2}$$

where A was the total area sampled and N the total number of particles, both parameters being included in the data printout. The use of this relationship will be discussed later.

4.0 ANALYSIS OF DATA - METAL TEMPERATURE ESTIMATION

4.1 Estimation Based on Mean Particle Diameter

As noted in the introduction, the carbides present in low alloy steels are metastable both in terms of size and chemical composition and so undergo time-dependent thermally-activated changes under service conditions; this report is only concerned with the particle size changes.

Metastable carbides obey coarsening kinetics of the form

$$d_t^{\,n} - d_o^{\,n} = Kt \qquad (1)$$

where d_t is the mean particle size at time t, d_o is the initial mean size and K is the temperature-dependent coarsening rate. Carbides of the type M_3C, VC and M_2C present in low-alloy steels obey $t^{1/3}$ kinetics in the temperature range 500 - 700°C i.e. n = 3 in equation (1) /5/. Generally, $d_t \gg d_o$ (see Discussion) so that equation (1) reduces to

$$d_t^3 = Kt \qquad (2)$$

Williams (formerly with CEGB) has published data on the temperature dependence of the carbide coarsening rate K for two low alloy steels i.e. 2-1/4 Cr-1 Mo and 1/2 Cr-Mo-V, as shown in Figure 3 /5/.

The relationship has the form

$$K = Ko \exp^{kT} \qquad (3)$$

or
$$\ln K = kT + \ln Ko \qquad (4)$$

Using some extrapolation of the lines in Figure 3, the following relationships were determined to represent the data for 2-1/4 Cr-1 Mo and 1/2 Cr-Mo-V

$$\ln K = 5.99\times10^{-2}T + \ln 3.25\times10^{-42}\ cm^3h^{-1} \quad (2\tfrac{1}{4}\ Cr\text{-}1\ Mo) \qquad (5)$$

$$\ln K = 6.00\times10^{-2}T + \ln 8.35\times10^{-42}\ cm^3h^{-1} \quad (\tfrac{1}{2}\ Cr\text{-}Mo\text{-}V) \qquad (6)$$

The procedure for temperature estimation thus involves determination of K in equation (2), given the measured value of d_t, the mean particle diameter and t, the time in service (1.3×10^5 h). The K value is then used in equation 5 or 6 to determine the value of T, the effective metal temperature over the life to date of the sampled component.

Unfortunately no particle coarsening data have been found for the other low-alloy steels of interest in the present work i.e. 1-1/4 Cr-1/ Mo and 1% Cr-Mo-V, although some particle spacing data for 1% Cr-1/2 Mo have recently been published/6/ (see later). Accordingly, the Williams 2-1/4 Cr data were used for all the Cr-Mo boiler and piping components and the 1/2 Cr-Mo-V data for all the 1% Cr-Mo-V. Table II presents the K values and metal temperature estimates thus obtained.

4.2 Estimation Based on Interparticle Spacing

As part of a recently-completed EPRI project, Cane and co-workers in England have developed a mechanistic approach to residual life assessment of low-alloy steel components based, in part, on structural coarsening. In this approach, the interparticle spacing is used rather than the particle mean diameter but the kinetics are essentially of the same form/6/.

The relationship between temperature T and particle spacing λ_t (id) is expressed by

$$T = \frac{\ln \left\{\frac{\lambda_t^3}{t}\right\} - \ln C_o}{k_c} \qquad (7)$$

where C_o and k_c are the pre-exponential and exponential constants, respectively, for the temperature dependence of C, the interparticle spacing coarsening rate.

The necessary parameters are given for 1 Cr-1/2 Mo in /6/ and, using the $(\frac{A}{N})^{1/2}$ spacing values from Table I, temperature estimates for 1-1/4 Cr-1/2 Mo components were obtained using equation (7). These results are presented in Table III, together with the results obtained using the Williams particle coarsening data for 2-1/4 Cr-1 Mo, for comparative purposes.

Very recently, it has been brought to the authors' attention that the expression for interparticle spacing used in Cane's work was

$$id = \frac{1}{2} \left(\frac{A}{N}\right)^{1/2}$$

i.e. the spacing values should be halved/7/. Using this adjustment, the temperature estimates for 1-1/4 Cr-1/2 Mo components were redetermined and these results are also given in Table III. It is apparent that the results using the $\frac{1}{2} (\frac{A}{N})^{1/2}$ spacing values are more realistic than previously obtained. The choice of expression for interparticle spacing will be examined in the Discussion section later.

5.0 DISCUSSION

In general, the temperature estimations presented in Tables II and III seem fairly realistic and within design expectations. Given some unfamiliarity with the quantitative metallographic approach, this can only be regarded as encouraging. It is now appropriate, however, to look more closely at some aspects of the work to assess better the confidence which may be placed on the results and also to identify areas where more work is needed.

5.1 Use of Inappropriate Coarsening Data

As noted, coarsening data could only be found for 2-1/4 Cr-1 Mo and 1/2 Cr-Mo-V so these were used to analyze results on 1-1/4 Cr-1/2 Mo and 1% Cr-Mo-V components in addition to those for which the data were appropriate i.e. some Cr-Mo boiler components.

The principal alloying elements in these low-alloy steels, chromium, molybdenum and vanadium, are all carbide stabilizers so that it might intuitively be expected that the resistance to coarsening should increase with alloy content. Thus 2-1/4 Cr-1 Mo would be expected to coarsen more slowly than 1-1/4 Cr-1/2 Mo. The coarsening propensity may actually depend on complex interrelationships between alloy and carbon atoms both in particles and in solid solution so that this simplistic view may not be wholly adequate. Comparing Cr-Mo with Cr-Mo-V alloys is particularly difficult, though the data of Williams/5/, Figure 3 indicate lower coarsening rates for 2-1/4 Cr-1 Mo than for 1/2 Cr-Mo-V.

As an example from the present results, replicas number 1-3295-60 (2-1/4 Cr-1 Mo) and 1-4006-55 (1 Cr-Mo-V but analyzed using 1/2 Cr-Mo-V data) had similar mean particle diameter values (0.090 and 0.087 μm) and gave temperature estimates of 543°C and 525°C, respectively. Because of the likely interactive effects of the three alloying elements (Cr, Mo, V), it is not known whether the above difference of ~20°C may represent an upper limit for the coarsening variability of the low-alloy steels of interest.

At present, it would appear that temperature estimates based on data for a lower alloy (e.g. for 1 Cr-Mo-V using 1/2 Cr-Mo-V data) will err on the low side; conversely, estimates on a lower alloy (1-/4 Cr-1/2 Mo using 2-1/4 Cr-1 Mo data) will err on the high side. The extent of these errors cannot be confidently predicted until data become available for the other materials, especially 1-1/4 Cr-1/2 Mo and 1 Cr-Mo-V.

Although the coarsening data published by Williams have been used, it should be noted that some doubt has been expressed about its validity. Other workers within CEGB have questioned the coarsening rates quoted for 1/2% Cr-Mo-V/8,9/ while, more recently, Parker produced some data at OHRD in 2-1/4 Cr-1 Mo which suggested different coarsening kinetics to those of the Williams work/10/. Figure 4 shows the coarsening rate-temperature relationships determined by Williams and Parker; clearly, different activation energies are indicated.

Although these disparate findings are of some concern, it transpires that, for the temperature range of interest, predictions from the William and Parker relationships are very similar. Table IV shows values for the 2-1/4 Cr components determined using both lines from Figure 4; clearly the discrepancies in this temperature range are negligible, despite the fact that the two lines do not, in fact, cross until ~567°C, ~30°C above the range of predicted temperatures.

5.2 Significance of Original Particle Size

The general relationship for particle coarsening expressed by equation (1) involves the term d_o, the original particle diameter. With n = 3, as found relevant to carbide coarsening in low-alloy steels/5/, it is normal to neglect d_o since d_o^3 is assumed to be much smaller than d_t^3. This assumption can be examined using data from the present work. Thus, for Tables I and II, ballpark values for K and d_t are $\sim 10^{-20}$ cm^3 h^{-1} and 10^{-5} cm, respectively.

Rearranging equation (1),

$$d_t = (Kt + d_o^3)^{1/3} \qquad (8)$$

so that, given Kt (0.84×10^{-15} cm^3), d_t can be determined as a function of d_o.

Taking values of d_o from 0.02 to 0.5×10^{-5} cm and plotting the results as d_t vs log d_o, Figure 5 shows that d_t is essentially independent of d_o until d_o values exceed ~0.3 d_t; below this value, d_t is essentially dependent on the coarsening rate K and time in service. On this basis, therefore, it is not necessary to have knowledge of the original microstructure provided it can be assumed that mean particle size has at least tripled in service. The validity of this assumption should be tested by quantitative metallographic characterization of new material.

5.3 Interparticle Spacing Values

Italian workers have used interparticle spacing as a quantitative coarsening parameter in work involving transmission electron microscopy of thin foils from steam pipe samples/4/. The spacing value id was obtained from:

$$id = \left(\frac{V}{N}\right)^{1/3}$$

where V is the foil volume and N the number of particles contained in the foil.

Clearly, when using surface replicas, area rather than volume is involved so that, in the present work, the expression:

$$id = \left(\frac{A}{N}\right)^{1/2}$$

at first seemed appropriate. It was subsequently learned, however, that the above expression is valid only for a regular square array of particles and that, for a random dispersion, the correct value for id is $\frac{1}{2}\left(\frac{A}{N}\right)^{1/2}$ /11/; this is the expression used by Cane et al/6/.

Other expressions for particle spacing have been quoted by Sellars/12/. These are:

$$\text{(i) spacing} = \frac{1.18}{N_A^{1/2}}$$

$$\text{(ii) spacing} = \frac{1.7\ r}{f^{1/2}}$$

where N_A = number of particles per unit area, r is the particle radius and f is the volume fraction of particles. The second equation is for the case of uniform spherical particles.

It is informative to compare the spacing values given by the various expressions using input data from the present work. Using the following values:

$$A = 330\ \mu m^2,\ N = 8713,\ d_t = 5.23 \times 10^{-2}\ \mu m$$

and taking $r = \frac{d_t}{2}$ and $f = \frac{N\pi r^2}{A}$ (assuming volume fraction = area fraction), the following spacing values are obtained:

Expression	Spacing μm
$\left(\frac{A}{N}\right)^{1/2}$	0.195
$\frac{1}{2}\left(\frac{A}{N}\right)^{1/2}$	0.098
$\frac{1.18}{N_A^{1/2}}$	0.229
$\frac{1.7\ r}{f^{1/2}}$	0.186

It is apparent that both the expressions quoted by Sellars give spacing values in good agreement with the $(\frac{A}{N})^{1/2}$ value; the $\frac{1}{2}(\frac{A}{N})^{1/2}$ value is clearly the exception.

Unfortunately, the original references containing the derivation of the $\frac{1}{2}(\frac{A}{N})^{1/2}$ relationship are very ancient and not readily obtainable. It would appear, however, that the expression is based on an assumption of a random, non-uniform particle dispersion whereas the other expressions implicitly assume a regular array of particles. In the present work, a large deviation is associated with the mean particle diameter as determined by the image analysis (see Table I). This indicates that a range of particle sizes is present so that the implicit assumption of a uniform dispersion (as needed to use the $(\frac{A}{N})^{1/2}$ expression) may indeed be incorrect.

The choice of expression to use may not, in fact, be too critical since all give a quantitative measure of spacing whether or not the absolute value is theoretically correct. A more practical criterion may be the availability of other data for comparative purposes. Thus, as noted, Italian data on 2-1/4 Cr-1 Mo was based on a volume equivalent of $(\frac{A}{N})^{1/2}$; also, some very significant work by Hale/13/ to be discussed later was based on the same expression. The recent work of Cane et al/6/, however, has used the $\frac{1}{2}(\frac{A}{N})^{1/2}$ value so that, for present purposes, it appears that both expressions may have to be used, depending on which other work is being compared.

5.3.1 Mean Free Path

The interparticle spacing as defined by the various expressions is actually the mean centre-to-centre distance between particles. In terms of the effect of particles on creep strength i.e. resistance to dislocation motion, a more meaningful parameter should be the mean free path between particles.

An expression for the mean free path in terms of area is

$$\left[\frac{A - N\pi\frac{\bar{d}_t^2}{4}}{N}\right]^{1/2}$$

where $N\pi\frac{d_t^2}{4}$ is the total area occupied by particles.

Using the same values for A, N and d_t as in Section 5.3, we obtain a mean free path value of 0.189 μm. This is not significantly different from the

spacing given by $(\frac{A}{N})^{1/2}$ and by the two Sellars expressions. This may offer some justification for using one of these expressions rather than the $\frac{1}{2}(\frac{A}{N})^{1/2}$ expression. From the values used, the ratio of d_t to spacing was ≈ 1:4, thus it appears acceptable to use spacing values rather than mean free path provided d_t < ≈1/3 of the spacing value.

5.4 Possible Error in Particle Spacing Value

It is worth noting the possibility of an error in the N value obtained using carbon extraction techniques. Thus, on a given surface, not all the carbides will be extracted, whether because of inadequate etching or simply because the portion exposed is smaller than the remainder below the surface. On the other hand, excessive etching may detach samples which, strictly speaking, belong on a lower plane of the sample. In general, however, incomplete rather than excessive collection would seem to be more likely so that the N value obtained would err on the low side; consequently the spacing values derived would err on the high side. In principle, this problem could be reduced by using thin foils and determining spacing from the mean volume per particle. A similar though less serious problem could arise, however, in connection with particles at the foil surface. Because of this error potential, the mean particle diameter would seem to be a more reliable index of coarsening than particle spacing.

5.5 Temperature Estimation Using Interparticle Spacing

From Table III, the temperatures estimated for 1-1/2 Cr-1/2 Mo components using the Cane et al model and spacings determined from $(\frac{A}{N})^{1/2}$ appeared excessive by about 30°C. Using the $\frac{1}{2}(\frac{A}{N})^{1/2}$ values, however, the estimates dropped to credible values and were mostly slightly lower than those obtained from the Williams particle coarsening data. Since, as noted in Section 4.1, the Williams estimates may have erred in the high side (due to use of 2-1/4 Cr-1 Mo data), the Cane estimates may be quite good. It has been learned, however, that the coarsening constants used in the Cane paper were early estimations and more accurate values may be forthcoming/7/.

The previously referenced Italian work/4/ contains particle spacing data on 2-1/4 Cr-1Mo and it is interesting to note that significantly different spacing values were found in bainitic and ferritic regions of the structures e.g. 0.42 μm for bainitic and 0.26 for ferritic. It is not known whether this bainitic-ferritic variation was considered by Cane and co-workers/6/. In obtaining the present quantitative data, no attempt has been made to select particular microstructure regions except, as noted earlier, to avoid grain boundaries and weld heat-affected regions.

5.6 Relating Interparticle Spacing to Creep Strain

Although the immediate aim of the quantitative metallographic work is the estimation of effective metal temperature, a longer term and intriguing possibility is to relate quantifiable microstructural parameters to actual creep deformation. Work done by Hale some 30 years ago at the National Physical Laboratory (UK) has already been noted briefly/13/ but it is now worthwhile to review some aspects in more detail.

Part of Hale's work involved quantitative microstructural characterization of ex-service 2-1/4 Cr-1 Mo superheater material using extraction replication; he obtained interparticle spacing data using the same $(\frac{A}{N})^{1/2}$ approach as adopted in the present study. A second, very significant part of the work was to take long-term creep test specimens and obtain spacing values as a function of known creep strain. These results are shown in Figure 6(a) and (b) In 6(a) creep strain is plotted against interparticle distance while in 6(b) the creep strain rate is plotted. It is interesting to note that the creep rate increases between 0.4 and 0.5 μm; at this stage, Figure 6(a) indicates a very low strain, ~0.1%.

The spacing increased perceptibly beyond this level of strain and the subsequent increase appeared independent of test temperature. In fact, Hale presented evidence to suggest that specimens had the same microstructure for a given strain irrespective of test temperature. This is not to imply that creep and structural changes are independent of temperature, however, but rather to emphasize the time-temperature dependency. Thus, what Hale showed was that similar structures resulted at the same strain in short-term high temperature specimens as in longer-term specimens tested at lower temperatures. The great importance of this observation is in terms of validating the use of overtemperature acceleration in service-simulated laboratory tests.

By relating microstructures to position on the creep curve, it appeared to Hale that, for 2-1/4 Cr-1 Mo, secondary creep continued (implying that tertiary was delayed) as long as fine needle-like particles were present and their spacing did not exceed ~0.5 μm. After this, tertiary creep ensued and rupture occurred when the spacing was ~1.2 μm.

Examining the present results on 2-1/4 Cr-1 Mo samples (see Table I), it is striking to find spacing values in the range 0.40 to 0.45 μm i.e. the values are similar to those found by Hale and are in the range where accelerating creep was beginning in the uniaxial specimens from which his data were obtained.

The electron micrographs on these specimens showed the presence of some needle-like particles - Figure 7 shows some typical examples. The density of these particles is not large, however, which, together with the spacing data, would suggest, from Hale's work, that the onset of tertiary creep in these

regions might be approaching. This is not to suggest any possibility of premature failure but it would certainly be worthwhile to re-examine these sampled regions after say 200 000 h.

Incidentally, the "doughnut" features in these micrographs are artifacts, believed to be associated with abnormal behaviour of the acetate tape used for replication. These features were edited out during the image analysis so did not affect the data.

Despite the obvious significance of Hale's work, there appears to have been little follow-up; clearly this is an area where further research could be usefully directed.

The model proposed by Cane et al/6/ has been considered in the context of temperature estimation but it also provides the potential means of predicting creep behaviour. The approach is based on the Dorn creep rate relation

$$\dot{\varepsilon} = A\sigma^n \qquad (9)$$

where $\dot{\varepsilon}$ = strain rate, A is the solid solution strength level, σ the applied stress and n the stress exponent.

Using the concept of effective stress and invoking particle coarsening kinetics, the following expression is obtained:

$$\dot{\varepsilon} = A_o \exp k_a \cdot \left(\sigma - \frac{\alpha' \mu b}{\lambda_t}\right)^n \qquad (10)$$

where A_o and k_a are material constants, T is the temperature, °K, μ is the shear modulus, b is the Burger's vector, λ_t is the measured particle spacing and α' is a particle/dislocation interaction parameter.

Using the values for 1 Cr-1/2 Mo given by the Cane paper for A_o, k_a, α', μ, b and n, equation (10) can be applied to data from the present work to obtain an estimate of the creep rate $\dot{\varepsilon}$ at the time of sampling.

For the main steam bottle header (replica 1-3245-114), the following data were found: $\lambda_t = \frac{0.36}{2}$ µm (to comply with Cane's use of $\frac{1}{2}\left(\frac{A}{N}\right)^{1/2}$); T = 798 K; σ = 48.5 MPa.

The other parameter values are as follows/6/:

$A_o = 4.2 \times 10^{-35}$ MPa^{-6}s^{-1}; $k_a = 5.30 \times 10^{-2}$ K^{-1}; $\mu = 63.2 \times 10^3$ MPa; $b = 0.245 \times 10^{-3}$ μm; $\alpha' = 0.228$; $n = 6$.

With the above values input to equation (10), we obtain.

$$\dot{\varepsilon} \sim 2 \times 10^{-8} \ \% \ h^{-1}$$

Clearly, this is an extremely low strain rate, suggesting that the component has so far sustained no significant creep strain and is not yet beyond the "threshold" region suggested by Hale's work in Figure 6(a).

From equation (10), a minimum particle spacing below which no creep occurs can be derived, as follows:

$$\sigma - \frac{\alpha' \mu b}{\lambda_t} = 0$$

so that $$\lambda_t \leq \frac{\alpha' \mu b}{\sigma}$$

Using the previous input values, we obtain $\lambda_t \leq 0.07$ μm. Even with a factor of 2 increase (due to use of the $\left(\frac{A}{N}\right)^{1/2}$ relationship) is is apparent that this "threshold" spacing is ~2.5 below the λ_t values determined for the 1-1/4 Cr-1/2 Mo samples. Thus, some creep is predicted through, as shown, its extent is virtually negligible.

The Cane et al approach is interesting and may offer the prospect of life prediction from microstructural observations. Clearly, however, a considerable amount of experimental work and/or plant data collection is needed to verify the model.

6.0 SUMMARY AND CONCLUSIONS

1. Techniques for obtaining quantitative metallographic data from surface replicas taken from low-alloy steel boiler, turbine and piping locations have been described.

2. Effective metal temperature at the sampled location can, in principle, be derived using the mean diameter or spacing of carbide particles in conjunction with coarsening kinetics data.

3. Particle coarsening data have only been found for 2-1/4 Cr-1 Mo and 1/2 Cr-Mo-V; recent work has also provided some data on spacing for 1 Cr-1/2 Mo.

4. Temperature estimates on Lakeview Unit 1 components gave credible results despite, in some cases, being obtained using inappropriate coarsening data. e.g. using 1/2 Cr-Mo-V data on 1 Cr-Mo-V samples.

5. Generally, it appears that temperature estimations based on data for a lower alloy (e.g. for 1 Cr-Mo-V using 1/2 Cr-Mo-V data) will err on the low side; conversely, estimates on a lower alloy (e.g. for 1-1/4 Cr-1/2 Mo using 2-1/4 Cr-1 Mo data) will err on the high side. The extent of the errors cannot be determined at present.

6. Two methods for obtaining particle spacing have been used in published work and give values differing by a factor of 2. Apparently excessive temperature estimates obtained using present data with a recently published model were found to be due to this difference.

7. It appears that, provided the particle size/spacing has at least tripled in service, knowledge of the original microstructure is not required in making temperature estimates based on coarsening kinetics.

8. The possibility of estimating creep strain from quantitative microstructural data has been examined with reference to two published approaches, one empirical, one theoretical. Present data, when fitted to these studies, have yielded interesting results.

7.0 RECOMMENDATIONS FOR FUTURE WORK

1. A short-term need is to quantify representative microstructures of pre-service materials, primarily to determine the validity of the usual assumption that $d_t^3 \gg d_o^3$. It should be possible to address this matter in the near future.

2. Coarsening kinetics data are required for 1 Cr-Mo-V and 1-1/4 Cr-1/2 Mo. In principle, this need could be met by a fairly low cost research program based on accelerated ageing heat treatments and quantitative metallography.

3. The work of Hale/13/ and the recent theoretical model of Cane et al/6/ strongly indicate the possibility of relating microstructural parameters to creep strain. The potential thus exists for both temperature and strain estimation from a surface replica, a powerful combination in terms of life assessment.

4. An aspect of quantitative metallography which has not been discussed in this report is the chemical compositional changes which occur in the carbides of low alloy steels in elevated temperature service. As with size and spacing, these changes can be related to service history and, indeed, some workers regard this as a superior approach/14/. Also in common with the "geometric" approaches which have been facilitated by the modern image analysis systems, compositional changes can now be monitored fairly readily using modern microanalytical systems in

conjunction with electron microscopy. Some work in this area has been done by the authors using facilities available at the University of Toronto, it is hoped to extend this work in the near future.

5. A state-of-the-art review on Quantitative Metallography related to Residual Life is currently being prepared for the Canadian Electrical Association by one of the authors (H.J.W.). It is likely that this will recommend a major research initiative in this area. Bearing in mind the typical lead-time for obtaining CEA funding, however, it is recommended that an in-house initiative be taken in the immediate future. This should involve formulation of a specific research program together with a commitment of the appropriate funding and manpower resources.

8.0 ACKNOWLEDGEMENTS

The authors acknowledge the efforts of A. Cervoni, T. Ryans, D. Limoges and M. Hooper in producing the surface replicas. Also appreciated is the work done by Mrs. A.J. Terry of ORF in developing image analysis techniques and for producing the required data.

Finally, discussions with colleagues, particularly Dr. M.A. Clark and Dr. D.V. Leemans have been very helpful.

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TABLE 1

QUANTITATIVE METALLOGRAPHIC DATA - LAKEVIEW UNIT 1

Sample	Replica ID	Material	Particle Mean Dia±Deviation (μm)	Median Particle Dia (μm)	Mean Inter-particle Spacing (μm)
A - BOILER COMPONENTS					
MS Bottle Header #1	1-3245-123	1.1/4 Cr-1/2 Mo	0.063±0.063	0.041	0.34
MS Bottle Header #10	1-3245-114	1.1/4 Cr-1/2 Mo	0.069±0.0675	0.05	0.36
MS Feeder Pipe #1	1-3245-123	1.1/4 Cr-1/2 Mo	0.0812±0.108	0.049	0.54
MS Feeder Pipe #10	1-3245-114	1.1/4 Cr-1/2 Mo	0.066±0.074	0.0434	0.34
SSH Outlet Header,East,Area B	1-3295-60	2.1/4 Cr - 1 Mo	0.090±0.108	0.055	0.45
SSH Outlet Header,West,Area B	1-3295-62	2.1/4 Cr - 1 Mo	0.0728±0.0589	0.0589	0.40
RH Outlet Header,East,Area A	1-3297-56	2.1/4 Cr - 1 Mo	0.079±0.069	0.0589	0.45
RH Outlet Header,Centre,Area B	1-3297-57	2.1/4 Cr - 1 Mo	0.0785±0.0796	0.0532	0.42
B - TURBINE COMPONENTS					
East Steam Chest, Main Steam	1-4006-55	1% Cr-Mo-V forging	0.087±0.093	0.059	0.29
East Steam Chest, Reheat	1-4006-52	1% Cr-Mo-V casting	0.084±0.082	0.058	0.35
HP Loop Pipe RHS	1-4030-5	1% Cr-Mo-V	0.106±0.103	0.067	0.39
HP Loop Pipe LHS	1-4030-10	1% Cr-Mo-V	0.099±0.0799	0.075	0.32
HP Inner Cylinder,Top Half Hot End	1-4031-50	1% Cr-Mo-V	0.0697±0.0624	0.0533	0.18

TABLE 1 (Contd)

QUANTITATIVE METALLOGRAPHIC DATA-LVGS UNIT 1 (Contd) (Page 2)

HP Inner Cylinder Top Half-Cold End	1-4031-49	1% Cr-Mo-V	0.0702±0.0695	0.0524	0.19
HP Rotor - Hot	1-4033-73	1% Cr-Mo-V	0.094±0.0868	0.064	0.30
HP Rotor - Cold	1-4033-75	1% Cr-Mo-V	0.11±0.107	0.0728	0.28
IP Rotor - Hot	1-4041-34	1% Cr-Mo-V	0.107±0.127	0.0659	0.27
IP Rotor - Cold	1-4041-32	1% Cr-Mo-V	0.052±0.05	0.0398	0.19
IP Loop Pipe,RHS	1-4035-14	1% Cr-Mo-V	0.0727±0.0524	0.0613	0.21
IP Loop Pipe,LHS	1-4035-13	1% Cr-Mo-V	0.0671±0.0495	0.0557	0.27
HIGH ENERGY PIPING					
Main Steam, Area 4	1-3245-45	1.1/4 Cr-1/2 Mo	0.124±0.116	0.086	0.47
Main Steam Y Pipe Leg,Area 2	1-3245-41	1.1/4 Cr-1/2 Mo	0.090±0.096	0.057	0.42
Reheat Y Pipe Area 2	1-3247-42	1.1/4 Cr-1/2 Mo	0.086±0.088	0.056	0.34
Reheat Y Pipe Leg, Area 4	1-3247-46	1.1/4 Cr-1/2 Mo	0.094±0.079	0.073	0.34

METAL TEMPERATURE ESTIMATIONS FROM QUANTITATIVE METALLOGRAPHY

Sample	Replica ID	Material	Temperature (°C)	Method
A - BOILER COMPONENTS				
MS Bottle Header #1	1-3245-123	1.1/4 Cr-1/2 Mo	525	
MS Bottle Header #10	1-3245-114	1.1/4 Cr-1/2 Mo	530	Williams-particle diameter
MS Feeder Pipe #1	1-3245-123	1.1/4 Cr-1/2 Mo	538	data for 2.1/4 Cr-1 Mo
MS Feeder Pipe #10	1-3245-114	1.1/4 Cr-1/2 Mo	528	
SSH Outlet Header,East,Area B	1-3295-60	2.1/4 Cr - 1 Mo	543	Williams-particle diameter
SSH Outlet Header,West,Area B	1-3295-62	2.1/4 Cr - 1 Mo	533	data for 2.1/4 Cr-1 Mo
RH Outlet Header,East, Area A	1-3297-56	2.1/4 Cr - 1 Mo	537	
RH Outlet Header,Centre, Area B	1-3297-57	2.1/4 Cr - 1 Mo	537	
B - TURBINE COMPONENTS				
East Steam Chest, Main Steam	1-4006-55	1% Cr-Mo-V forging	525	Williams-particle diameter
East Steam Chest, Reheat	1-4006-52	1% Cr-Mo-V casting	523	data for 1/2 C-M-V
HP Loop Pipe RHS	1-4030-5	1% Cr-Mo-V	523	
HP Loop Pipe LHS	1-4030-10	1% Cr-Mo-V	534	

TABLE 2 (contd)

METAL TEMPERATURE ESTIMATIONS FROM QUANTITATIVE METALLOGRAPHY (Contd) (Page 2)

HP Inner Cylinder,Top Half Hot End	1-4031-50	1% Cr-Mo-V	514	
HP Inner Cylinder Top Half-Cold End	1-4031-49	1% Cr-Mo-V	514	Williams-particle diameter
HP Rotor - Hot	1-4033-73	1% Cr-Mo-V	528	
HP Rotor - Cold	1-4033-75	1% Cr-Mo-V	536	data for 1/2 C-M-V
HP Rotor - Hot	1-4041-34	1% Cr-Mo-V	535	
LP Rotor - Cold	1-4041-32	1% Cr-Mo-V	499	
LP Loop Pipe,RHS	1-4035-14	1% Cr-Mo-V	516	
LP Loop Pipe,LHS	1-4035-13	1% Cr-Mo-V	511	
HIGH ENERGY PIPING				
Main Steam, Area 4	1-3245-45	1.1/4 Cr-1/2 Mo	521	
Main Steam Y Pipe Leg,Area 2	1-3245-41	1.1/4 Cr-1/2 Mo	543	
Reheat Y Pipe Area 2	1-3247-42	1.1/4 Cr-1/2 Mo	541	Williams-particle diameter
Reheat Y Pipe Leg, Area 4	1-3247-46	1.1/4 Cr-1/2 Mo	546	data for 2.1/4 Cr-1 Mo

TABLE 3

1.1/4 Cr-1/2 Mo COMPONENTS - TEMPERATURE ESTIMATES

Component	Cane Estimate[1] (°C) A	B	Williams Estimate[2] (°C)
MS Bottle Header #1	557	522	525
MS Bottle Header #10	560	525	530
MS Feeder Pipe #1	580	545	538
MS Feeder Pipe #10	557	522	528
MS Area 4	573	539	521
MS Y Area 2	567	533	543
RH Y Area 2	557	522	541
RH Y Area 4	557	522	546

[1] From particle spacing using parameters for 1 Cr - 1/2 Mo

[2] From mean particle diameter using data for 2.1/4 Cr - 1 Mo.

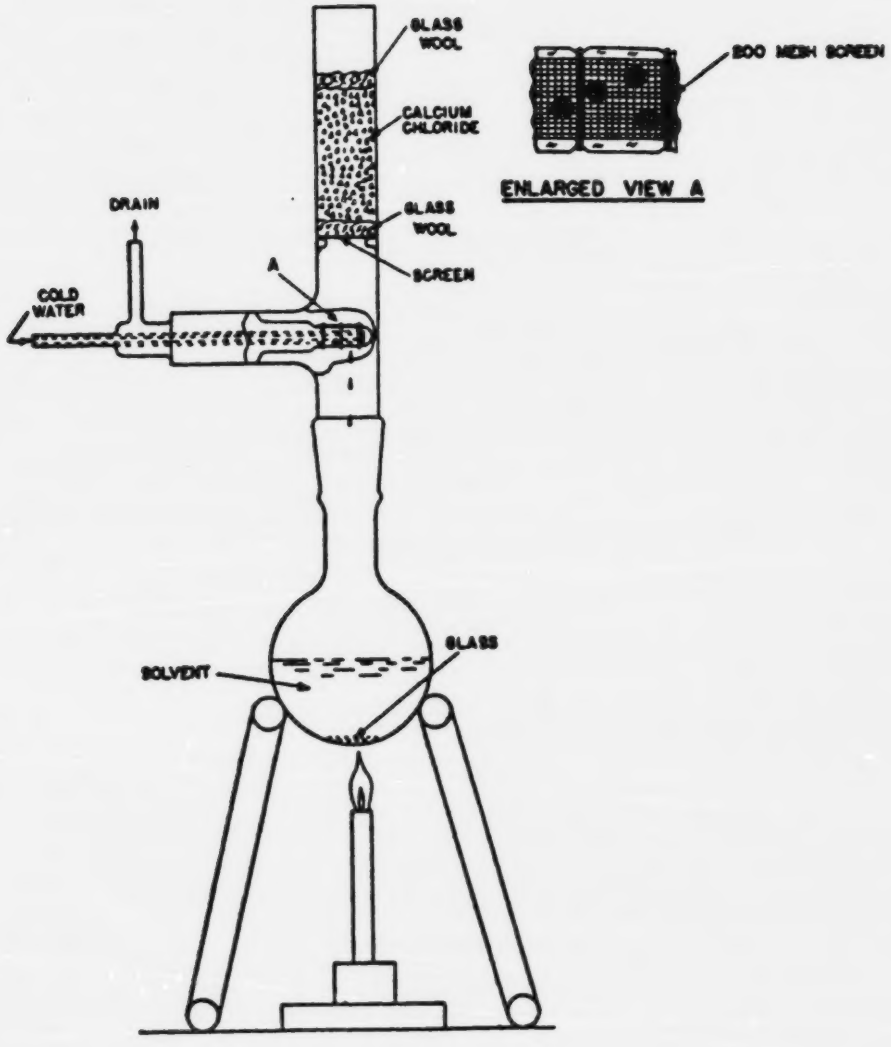
Cane estimates A - using $id = \left(\frac{A}{N}\right)^{1/2}$

B - using $id = 1/2 \left(\frac{A}{N}\right)^{1/2}$

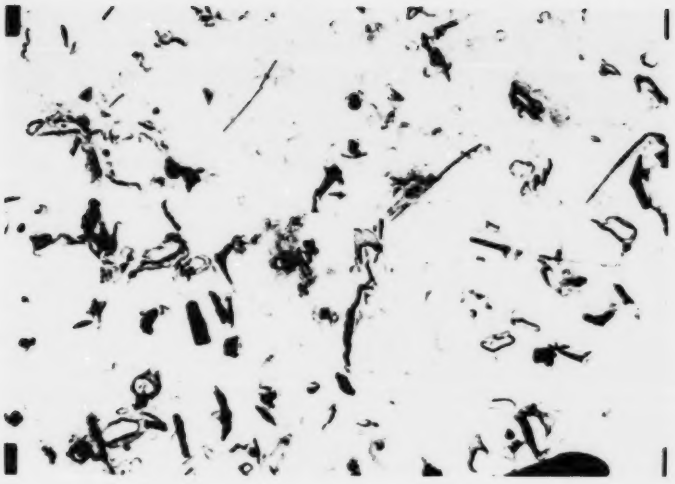
TABLE 4

TEMPERATURE ESTIMATION FOR 2.1/4 Cr - 1 Mo COMPONENTS USING WILLIAMS AND PARKER DATA

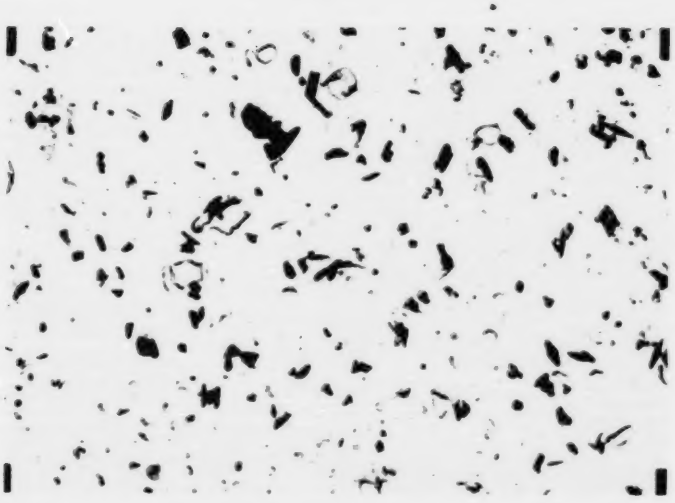
Component	Williams (°C)	Parker (°C)
SSH Outlet Header, E, Area B	543	544
SSH Outlet Header, W, Area B	533	530
RH Outlet Header, E, Area A	537	535
RH Outlet Header, C, Area B	537	537



1 Replica Washing Apparatus

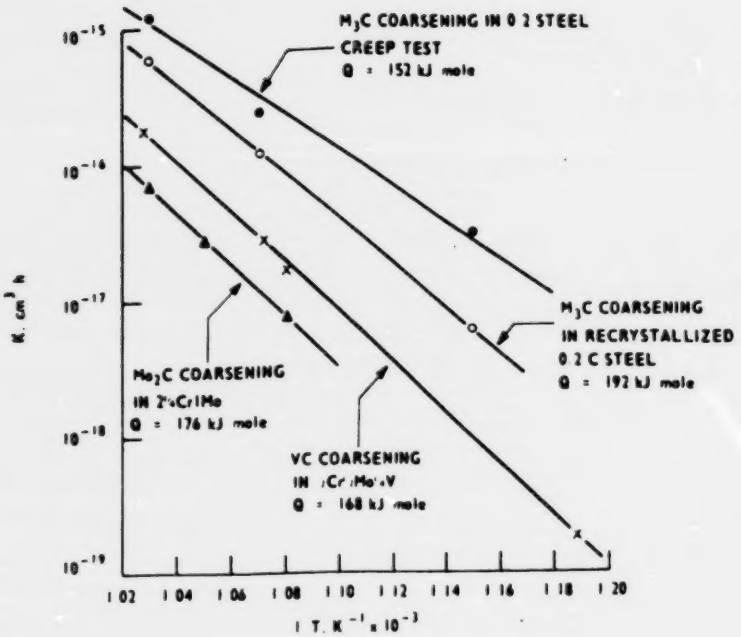


HP INNER CYLINDER, UNIT 4
1/2 Cr-Mo-V X 10,000

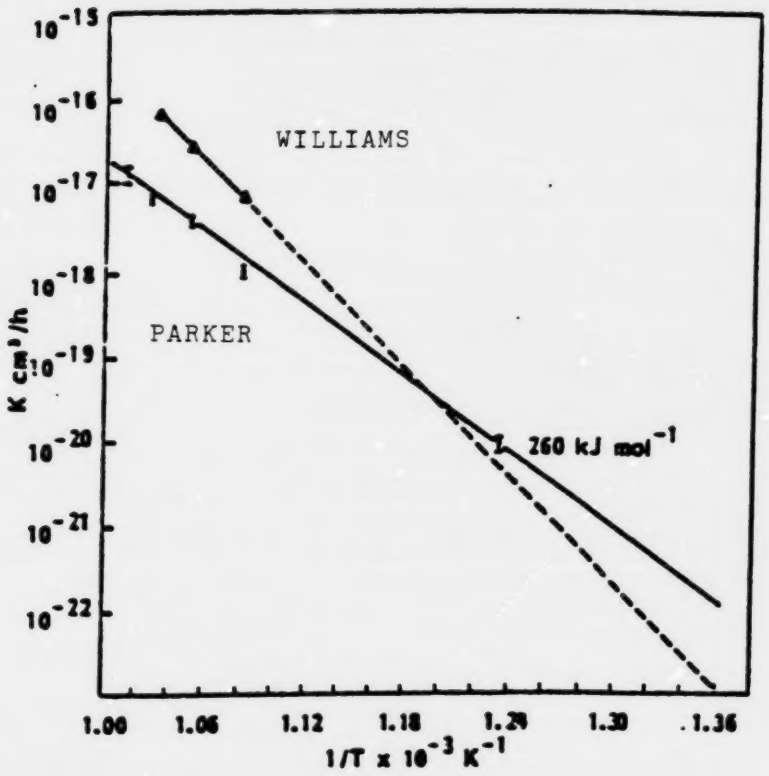


HP ROTOR, UNIT 4
1/4 Cr-Mo-V X 10,000

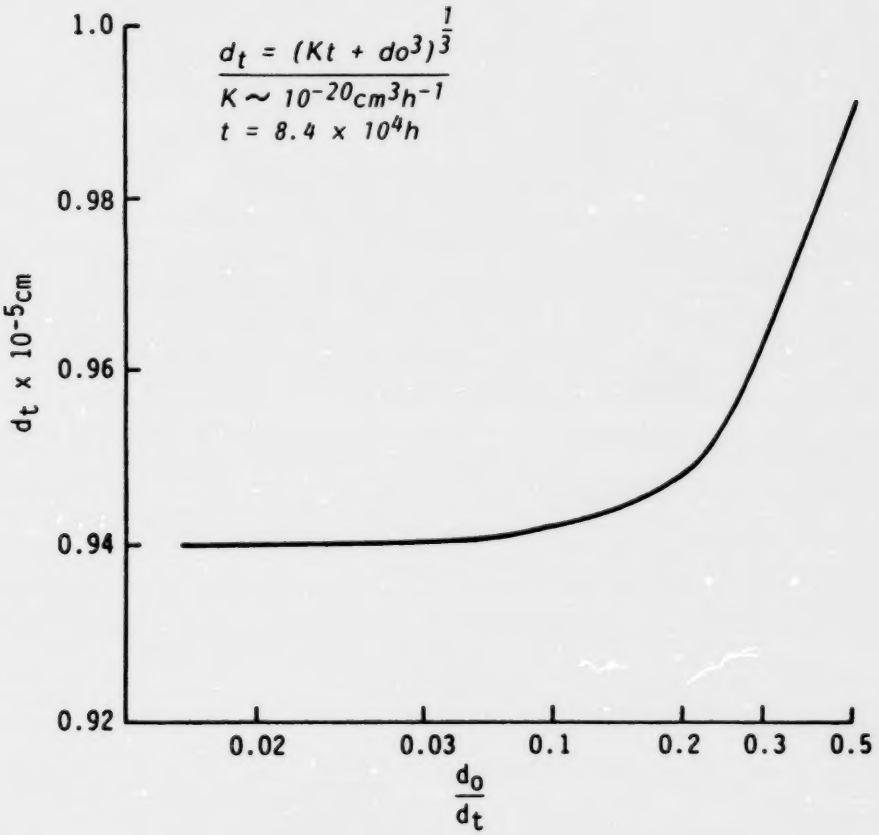
2 **Typical T.E.M. Micrographs**



3 Williams Coarsening Rate Data

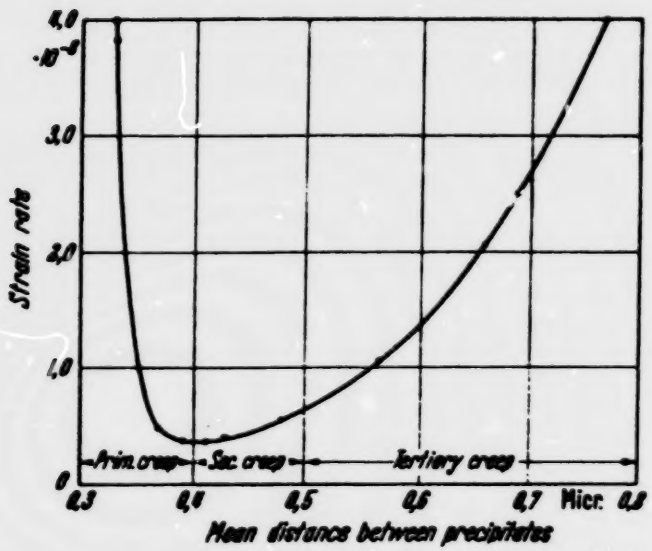


4 Comparison of Williams and Parker Coarsening Results



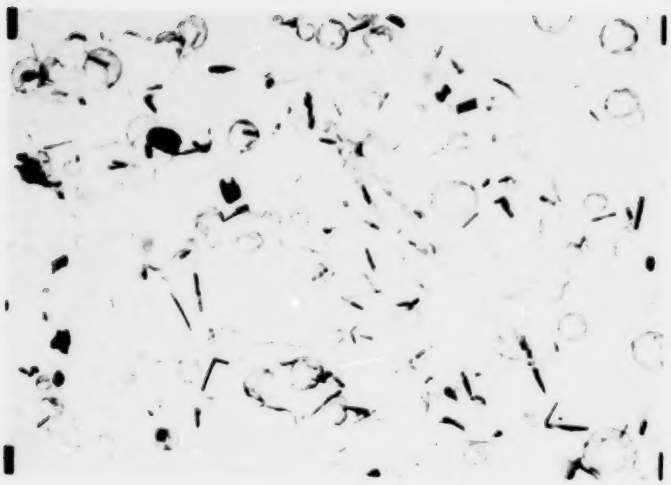
5 Plot Showing Sensitivity of d_t to d_o

Change in mean distance between precipitates with increasing strain for creep tests at temperatures between 570° and 650° C



Strain rate during creep test at 650° C plotted against mean distance between precipitates

6 Hale Results Relating Particle Spacing to Creep (a) Creep Strain, (b) Creep Strain Rate



SSH HEADER, UNIT 4
Area 10, X 10,000

SSH HEADER STUB TUBE
Area 10, X 10,000

7 **Remaining Needle-like Particles in 2-1/4 Cr-1Mo Samples**

APPENDIX

RESEARCH

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MATERIALS DIVISION

Inorganic Materials Centre

CHARACTERIZATION OF CARBIDES IN LOW-ALLOY STEEL REPLICAS USING IMAGE ANALYSIS TECHNIQUES

Image Analysis Services
Report No. 1

A.J. Terry
14 July, 1987

for

Ontario Hydro Research
800 Kipling Avenue
Toronto, Ontario
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Attention: Dr. H.J. Westwood

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INTRODUCTION

Ontario Hydro has approached Ontario Research Foundation (ORF) to seek technical assistance in developing the methodology in performing the characterization work involved in quantifying the dispersed carbides within the grains of low-alloy steels in power plant components. Image analysis techniques have been developed for the characterization.

Samples in the form of carbide replicas have been provided by Ontario Hydro. Data obtained by image analysis will point out any changes that may have occurred over the years and will provide a set of comparison statistics for sampling that will take place in the future.

A description of the project was outlined in ORF Proposal P 5473-CI and has been initiated as ORF Account No. 66-10777 entitled "Carbide Characterization". Work was begun in May 1987 and is expected to continue into 1988.

METHOD

The samples provided by Ontario Hydro have been in the form of replicas of the carbides photographed on the scanning* electron microscope (SEM) at a magnification of 10,000X. Each photograph was placed on the macro-viewer and the image was transferred via video camera to the television screen of the LeMont OASYS Optical Analysis System which operates on a DEC 11/73 computer. Six photographs have been provided for each sample so that 3,000 to 8,000 carbides could be characterized. A size distribution of about 1500 particles is generally accepted as having sufficient data to provide a 95% confidence level that does not change.

The television screen of the image analyser has a grid of 512 by 480 picture points (pixels). Up to 256 grey levels may be assigned to features and up to 15 of these classes may be analysed at any time. In this case, the carbides were assumed to be the darkest features and the background a lighter level. Any features with a grey level below a set value would have the picture points turned "on" and so would be included in the analysis.

* for scanning, read transmission

The screen of the image analyser was calibrated prior to each set of analysis by projecting the image of a ruler onto the screen and determining the number of picture points per unit length. The 10,000X magnification of the photographs was then figured in.

The quality of the images was enhanced using the image editor. The fine carbides were more readily detected after small feature enhancement which is a filter that operates on a 7X7 matrix. This amplifies the grey level at locations of rapid change. This function was designed to increase the contrast of a light field containing many dark dots. The background noise increases with this option so it was followed by a filter which is a spatial processing operation involving a 3X3 array of numbers. This pixel mask operates simultaneously on a small group of pixels to produce a new single pixel. The value of this single pixel then depends not only on the value of its corresponding pixel in the original image, but on all the pixels surrounding it in the matrix. After filtering, each pixel in the new image is the weighted average of nine pixels in the original image: the pixel corresponding to the new one together with its 8 nearest neighbours.

Interactive editing was also performed in some cases. This involves the use of a light pen on a digitizing tablet to draw a boundary between particles which were obviously touching and should be separated.

Some photographs had shaded areas where the background grey level varied. A "joybox" could be fit around only the area of interest so that a smaller field of view was presented for analysis. Or if the area to be deleted occurred in the middle of the photograph, it could be outlined with the digitizing pen, coloured at a level not being used for analysis and be excluded from the results.

One set of samples was received in a different form. A thesis by Dr. Latif had reproductions of SEM photographs from which analyses were required. In this case the photographs were simply photocopied and the copies were used as the samples following the same procedures as described above. A maximum of three photographs per sample were analysed for this group.

Representative Polaroid photographs were provided for each type of image enhancement that was used. A photograph of the original image in black and white and the enhanced image in colour were taken to portray the techniques used.

After the enhanced image was ready for analysis it was then processed by the computer to obtain specific measurements of interest from the menu of available parameters.

The size distribution data were based on the equivalent circular diameters of the particles. The numbers of particles in each of twenty size classes (0 to 0.4 micrometre in steps of 0.02 µm) were provided for each sample in the form of a histogram. The smallest size class was actually 0.018 to 0.02 µm, as 0.018 µm was the finest particle which could be resolved using this technique. In addition, a large bin at the top end of the size classes was added (0.4 to 1.0 µm) in order to catch any larger features. The mean and standard deviation were also provided for each size distribution. The mean is expressed as the average on the printout and is the arithmetic mean of the physical measurement from all particles. The deviation is the root mean square deviation of the physical measurement from all particles. The percent deviation is the ratio of the root mean square deviation divided by the average times 100. The median is the estimate of the value at which 50% of the particles are below the value presented. The most probable is the average of the upper and lower class limit in which the maximum population occurs. The median and most probable values are determined by interpolation on histogram data.

Most of the samples analysed had a size distribution curve that was not normal. Consequently, the spread of data could be described using Tchebysheff's Theorem: given a whole number K & a set of n measurements, at least $(1-1/K^2)$ of the measurements will lie within K standard deviations of their mean. This theorem is very conservative and applies to any distribution of measurements.

The interparticle spacings in micrometres were calculated from $(A/N)^{1/2}$ where N is the number of particles in area A.

SUMMARY OF PROGRESS OF WORK

To date, 27 sets of SEM micrographs of carbide replicas within the grains of low alloy steels have been characterized by image analysis. In addition, 8 samples from the Latif thesis have been analysed. The results are summarized in Appendix A for the average (including deviation) and median diameters and interparticle spacings for each set of samples. The detailed computer printouts and any photographs taken during the image analysis process have been supplied to Dr. Westwood.

The sample "LGS unit 1 main steam Y pipe area 2 (20,000X)" will be used as an example for describing the spread of data. In this case the average, or mean, ($\bar{y}$) is 0.21 µm and the deviation (s) is 0.13 µm. Using K-2 & 3 respectively, we can state that at least 75% of the measurments are in the interval 0.21 ± 0.26 µm and that at least 89% of the measurements are in the interval 0.21 ± 0.39 µm. If the distribution is normal the Empirical Rule could be used to state that 95% of the measurements are in the interval 0.21 ± 0.26 µm ($\bar{y} \pm 2s$).

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K.L. Yan, Ph.D.
Section Manager

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